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## The Influence of Casting Solution Composition and Stirring Conditions Against Mechanical Strength and Performance of Polyvinylidene fluoride (PVDF)-Polysulfone (PSf) Composite Membrane on Textile Industrial Wastewater Treatment.

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### ABSTRACT

Generally, this research aims to get a composite membrane that not only has the mechanical strength, a good thermal and chemical resistance, but also economically. More specifically, the purpose of this research is to know the influence of the casting solution composition and stirring condition against mechanical strength and performance of PVDF-PSf composite membrane on textile industrial wastewater treatment. In addition, this research is also directed to determine the morphology of the surface of PVDF-PSf composite membrane. In this research, the PVDF-PSf composite membrane made by phase inversion method with immersion precipitation techniques in 100% H<sub>2</sub>O as non solvent. To know the influence of the casting solution composition to mechanical strength and the performance of the membrane, the membrane casting solution composition varied in the range PVDF/PSf/NMP/NH<sub>4</sub>Cl (%w/w) 12/2/84/2-16/0/0/84. Meanwhile, to find out the effect of stirring condition on mechanical strength and the performance of the membrane, the membrane making on this research was conducted with the variation of the stirring temperature in the range of 40-60 °C. Measuring results of the PVDF-PSf composite membrane mechanical strength using autographs instruments shows that the PVDF-PSf composite membrane have mechanical strength in the range Of 908,49 N/m<sup>2</sup> – 2.118,09 N/m<sup>2</sup>. Flow rate (flux) measurement results of PVDF-PSf composite membrane using Dead end membrane reactor shows that the PVDF-PSf composite membrane have flux in the range of 1.200,38 L/m<sup>2</sup>.jam – 2.779,45 L/m<sup>2</sup>.jam. Meanwhile, the selectivity (rejection percentage) measurement results of PSf-PVDF composite membranes using Spectrophotometric UV-Visible instruments, mind that rejection coefficient of PVDF-PSf composite membrane is in the range of 89.88%-92.78%. While from the results of the surface morphology analysis of PVDF-PSf composite membrane using Scanning Electron Microscopy (SEM) instruments, note that casting solution affect the morphological structure of the membrane. The higher levels of the PSf that is added into the solution, porous membranes produced more rift. And vice versa. In addition, the analysis results of the PVDF-PSf composite membrane surface morphology using SEM showed that the stirring temperature effect on morphological structure of the membrane. The higher stirring temperature in the preparation of the PVDF-PSf composite membrane, the more tenuous porous structure resulting on the surface of the PVDF-PSf composite membrane. And vice versa.

**Keyword** : Membrane, PVDF, PSf, Phase Inversion, Immersion-Precipitation

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## INTRODUCTION

One of the widely used polymer material in the form of membranes for the purposes of textile wastewater treatment is polysulfon [1]. Polysulfon is known for its excellent characteristics, include: (1) a good solubility in polar aprotic solvents are a large number of (dimethylformamide (DMF), dimetilasetamida (DMAc), dimetilsulfoksida (DMSO), halogen derivatives, aniline, and nitrobenzene); (2) high thermal resistance (150-170 ° C), (3) a good chemical resistance almost on the entire range of pH and oxidative medium (hypochlorite 5-7%, hydrogen peroxide 3-5%), and (4) a good mechanical resistance (cracks, flexibility, torsion) [2]. However, the use of polysulfone commercial membranes are generally limited due to the symmetry structure of the material as well as a range of pore size and porosity are limited. The symmetry membrane has mass transfer resistance are much higher than those of asymmetry membrane. These conditions will increase the fouling potential on the surface and membrane internal pore of polysulfone. Decrease in the use of the membrane will go hand in hand with increased membrane fouling potential is concerned. Some methods to overcome this weakness has been made, including by conducting blending between polysulfone polymer with other polymer material who are capable of forming dense membrane with asymmetry structures [3]. Polyvinylidene fluoride (PVDF) has become the new polymer materials begin to widely used in some years due to his excellence in thermal resistance ( $\geq 300$  °C), chemically (good chemical resistance almost on the entire range of pH and oxidative medium) and had very good mechanical strenght ( $\pm 1,500-5,900$  N/m<sup>2</sup>), even better than the polysulfone membrane, as well as its ability to form an asymmetry membrane [4].

During this time, polysulfone and PVDF membranes have been successfully prepared by using three (3) kinds of methods, namely sintering, track etching, and also the phase inversion [5]. But this time, most of the commercial polysulfone and PVDF membrane produced with the phase inversion method, mainly because the process is more simple and had flexible production scale. Thus, the relatively low production cost can be maintained. This method is also considered as the main method of the PVDF membrane preparation [6].

Phase inversion can be described as the demixing process in which initial homogeneous polymer solution is transformed from liquid to solid form in a controlled condition [7]. This transformation can be achieved through a number of techniques, namely (a) thermal induced phase separation (TIPS); (b) the controlled solvent evaporation from the system; (c) precipitation from the steam phase and (d) the immersion-precipitation (IP). However, from a number of these techniques, TIPS and IP are the two most widely used techniques in the manufacture of polymer membrane, such as polysulfone and PVDF. While immersion-precipitation is a process by which a polymer solution is casted on the appropriate support solids and then soaked in a coagulation batch which containing non-solvent. The exchange of the solvent in the polymer solution with non-solvent from coagulation batch produces a phase inversion. Immersion-precipitation is a major technique in the manufacture of asymmetry membrane in the industry because the simple process [8].

Preparation of PVDF-PSf composite membrane with blending method will be able to solve two problems at once, where by blending polysulfone polymer on PVDF polymer, the membrane characteristics which produced, in addition to being better in terms of thermal resistance, chemical and mechanical strenght, will also have a much higher fouling resistance. In addition, because the polysulfone is a type of polymer which is more economical than PVDF, then the use of this material will be able to push the operational cost of wast water treatment Installations of textile industry.

## EXPERIMENTAL

### Materials

Materials research include: PVDF (MW 534 000, Sigma-Aldrich), NMP (anhydrous 99.5%, Sigma-Aldrich), NH<sub>4</sub>Cl ( $\geq 99.5\%$ , Sigma-Aldrich), polysulfone (Mw 35,000 (pellets), Sigma-Aldrich), deionized water, ethanol (C<sub>2</sub>H<sub>5</sub>OH) ( $\geq 99.9\%$ , Sigma-Aldrich). In the meantime, to conduct performance tests (flux and rejeksi) PVDF-PSf composite membrane, used batik industrial wastewater obtained from batik SMEC Sidoarjo, East Java, Indonesia.

## Equipment and Instrument

Research equipment includes: a watch glass, petri dish (d = 9 cm), spatulas, beaker glass, measuring cups, glass plate and magnetic stirrer. Beside that, in this experiment also used Fourier Transform Infra Red (FTIR), autograph, "Dead end" membrane reactor, and spectrophotometer (Shimadzu UV-2401-PC) instrument.

## Preparation of PVDF-PSf Composite Membrane

PVDF dissolved in NMP at a temperature of 40 °C. Casting solution composition was varied in the range (PVDF/NMP/NH<sub>4</sub>Cl) (% w/w) 12/84/2-16/84/0. NH<sub>4</sub>Cl is used as a pore-forming additive (porogen). Furthermore, 2 %(w/w) polysulfone is added to the PVDF membrane casting solution and then stirred using magnetic stirrer for 15 minutes. Polymer solution that formed subsequently casted on a petri dish and then soaked in a coagulation bath that contains non-solvent (distilled water) at a temperature of 40 °C to regardless of casting.

The solid membrane that resulting, further removed from the coagulation bath and rinsed with running water to remove residual solvent and NH<sub>4</sub>Cl from the membrane. The membrane is then dried and stored in glass sheets given load to prevent membrane shrinkage. The same procedure also applies to the preparation of PVDF-PSf composite membrane with the chitosan solution addition: 1.5; 1; and 0.5% (w/w), and also to the preparation of PVDF-PSf composite membrane with stirring temperature 50 and 60 °C.

## Depreciation of PVDF-PSf Composite Membrane

To determine the level of membrane shrinkage during the phase inversion process, membrane diameter measurements were taken before and after the phase inversion process. Membrane measurements performed on 4 (four) sides, includes: (a) the vertical, (b) the horizontal and (c) two sides of the diagonal.

## Characterization of PVDF-PSf Composite Membrane

To determine the physical characteristics (surface morphology and pore size) and mechanical strength of the membrane (resistance to stress and strain) also permeability, conducted an analysis of the PVDF-PSf composite membrane, with the following information: (a) the chemical compounds test using Fourier Transform Infra Red (FTIR) to ensure formation of composite membrane PVDF-PSf; (b) Imaging surface morphology and cross section of the membrane by scanning electron microscopy (SEM); (c) Measurement of membrane mechanical strength with autograph; and (d) measurement of membrane permeability (flux) with a "dead-end" membrane reactor.

## Permeability of PVDF-PSf Composite Membrane

The procedure is follows. At the bottom of the "Dead-end" reactor membrane, ie at the laying of the membrane, placed filter paper that was cut in the size of the membrane ( $\pm$  6 cm). Membrane then placed on the top of filter paper. After that, the "dead-end" membrane reactor strung back and 100 mL aquadestilata applied thereto as feed solution. At the same time, pressure of 1 atm of compressor flowed into the "Dead-end" membrane reactor. Obtained permeate is collected and recorded the time it takes until all the feed solution filtered out. Furthermore, based on the data of feed solution volume, membrane area and the time it takes until all the feed solution filtered out, the flux was calculated.

The measurement of PVDF-PSf composite membrane permeability on batik industrial wastewater treatment done with the same procedure with membrane permeability test above, except that the original feed solution aquadestilata replaced by the wastewater.

## Selectivity of PVDF-PSf Composite Membrane

Furthermore, to determine the rejection coefficient of the PVDF-PSf composite membrane in batik industrial wastewater treatment, the dissolved oxygen of permeate that obtained was measured and then compared with dissolved oxygen level of the feed solution (wastewater sample).

## RESULT AND DISCUSSION

### PVDF-PSf composite membrane preparation

In this research, the preparation of the PVDF-PSf composite membrane is done using phase inversion method, where the transformation from liquid state (casting solution) to solid state (membrane), induced by particle exchange processes between a non solvent particles with solvent (NMP) particles which contained in the casting solution. The solubility parameters differences between non-solvent and solvent which is used in the preparation of membrane, will increase the total heat of mixing ( $\Delta H$ ), which will automatically increase the Gibb's free energy ( $\Delta G$ ) of the reaction system. The increase of the Gibb's free energy will trigger the occurrence of coagulation of membrane casting solution so that it can generated the solid PVDF-PSf composite membrane. In figure 1 appears to the interaction that occurs between the membrane-forming components.

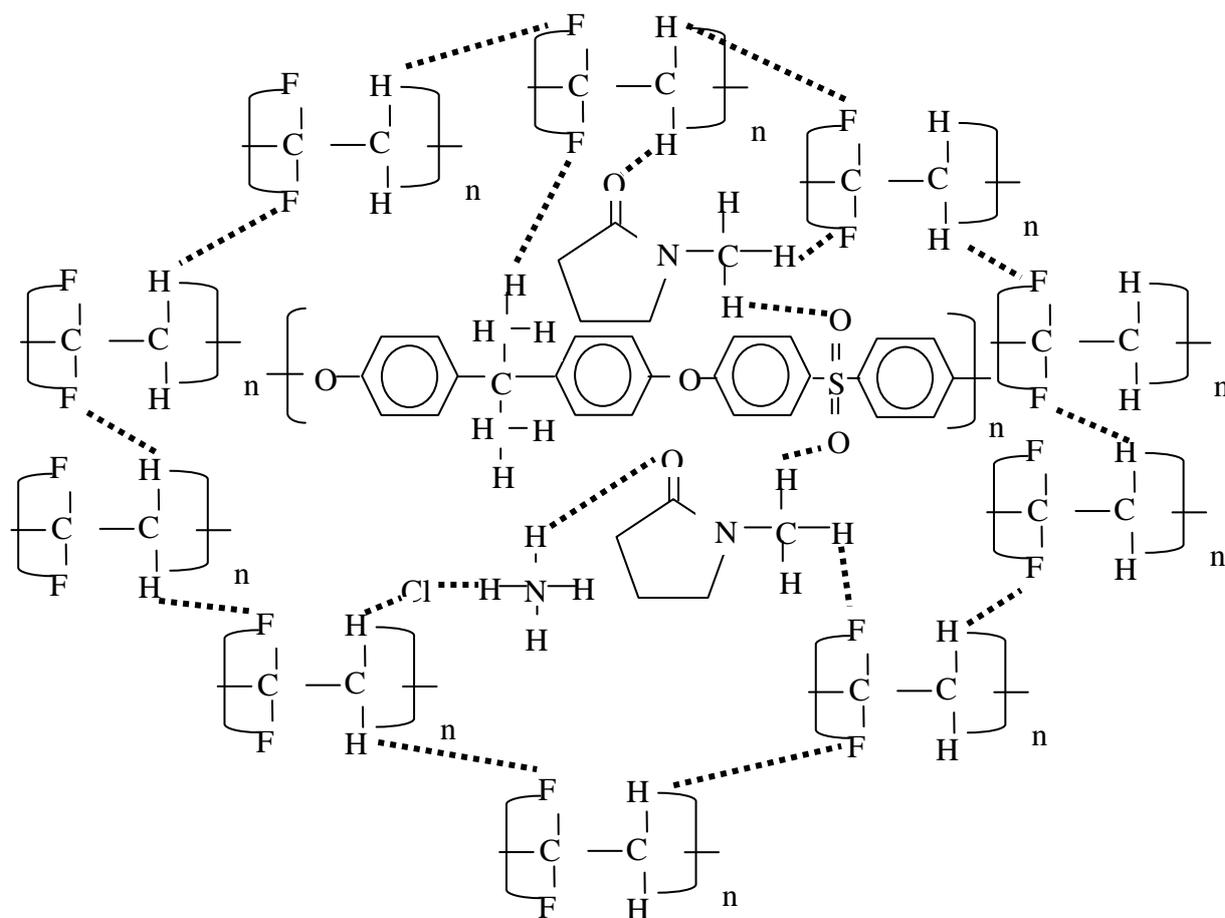


Figure 1: Interaction between the membrane forming component (hypothetic)

### Characterization of PVDF-PSf composite membrane

Characterization of the PVDF-PSf composite membrane is done by using four types of testing, including the chemical compounds test using FTIR to ensure formation of the PVDF-PSf composite membrane, the surface morphology test of the membrane using SEM to determine the pore structure is formed on the PVDF-PSf composite membrane test, pore size using the SAA, and mechanical strength test of membrane use autograf.

### The chemical structure test of PVDF-PSf composite membrane

Chemical structure of PVDF membrane and PVDF-PSf membrane composite that has been successfully prepared, characterized using FTIR. Figure 2 shows the infrared spectra of the PVDF membrane, PSf membrane

and PVDF-Psf membrane. Infrared Spectra based on PVDF membrane (a) which is obtained from the analysis using FTIR instruments, was detected PVDF molecule characters, namely (a) peak at wavenumbers  $1,401.15 \text{ cm}^{-1} - 1,382.99 \text{ cm}^{-1}$  indicating the presence of C-H alkane; and (b) peak at wavenumbers  $1,181.14 \text{ cm}^{-1}$  indicating the presence of C-F. While the Infrared Spectra data based on membrane polisulfon (b), the typical character of the polisulfon molecule was detected, namely: (a) peak at wavenumbers  $3,408.00 \text{ cm}^{-1}$  indicating the presence of  $\text{NH}_2$ ; (b) the peak at wavenumbers  $1,654.84 \text{ cm}^{-1}$  indicating the existence of C=C aromatic; and (c) peak at wavenumbers  $2,869.66 \text{ cm}^{-1} - 2,965.93 \text{ cm}^{-1}$  indicating the presence of alkane C-H/C-H aromatic. In the meantime, based on the Infrared Spectra of the PVDF-Psf composite membrane resulting from the manufacturing process with the stirring temperature  $40 \text{ }^\circ\text{C}$ , PVDF molecule character is detected, and the PSf, namely: (a) peak at wavenumbers  $3,373.46 \text{ cm}^{-1}$  indicating the presence of typical polisulfon  $\text{NH}_2$ . There is a shift in the peak of the spectra and composite membrane on PVDF-Psf indicated that one of the interactions between the particles of PVDF and PSf is through a primary amine molecules; (b) the peak at wavenumbers  $2,965.49 \text{ cm}^{-1}$  indicating the existence of - CH.

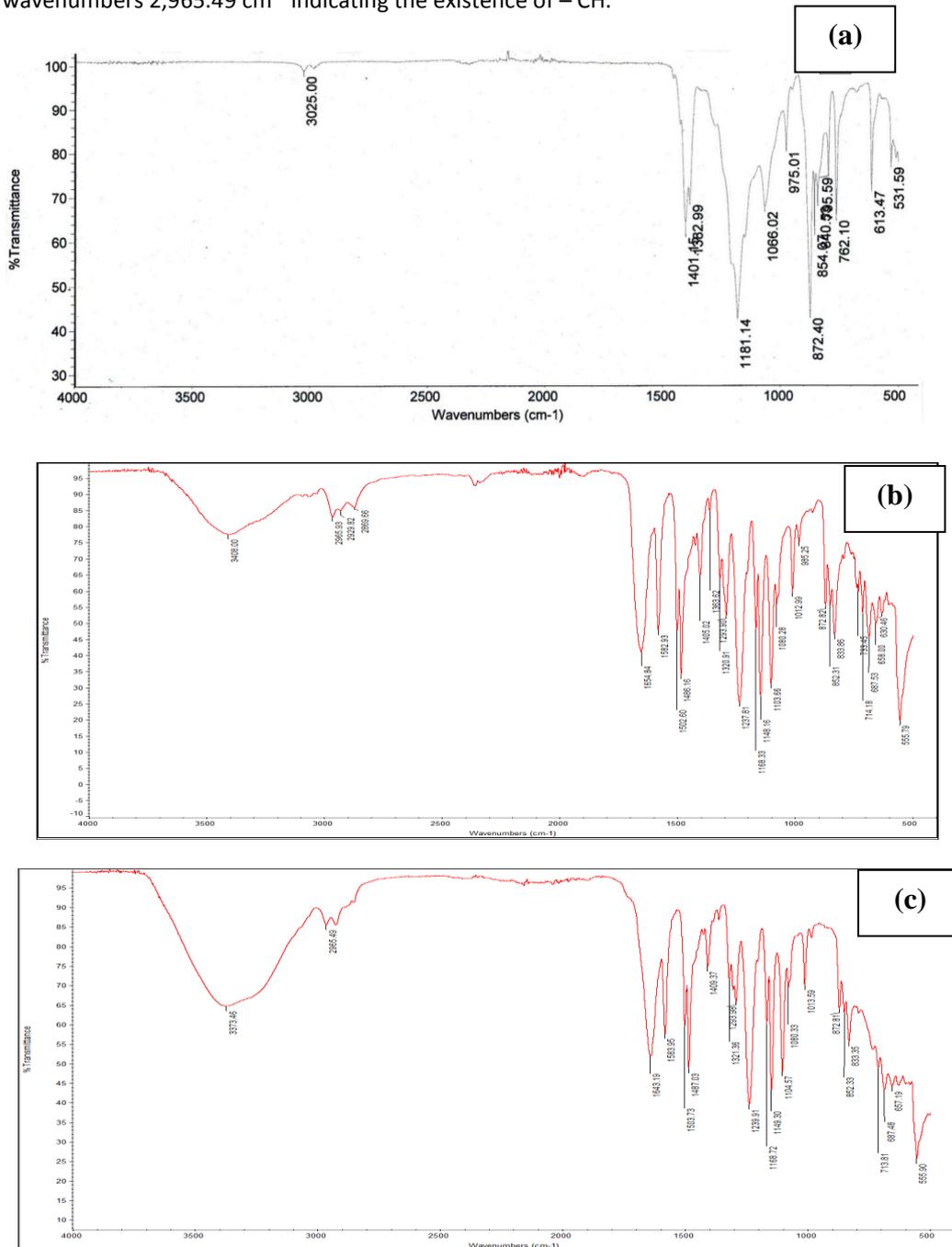


Figure 2: The IR spectra of : PVDF membrane (a); PSf membrane (b); and PVDF-Psf composite membran (c)

The surface morphology and cross section test of PVDF-PSf composite membrane by using SEM

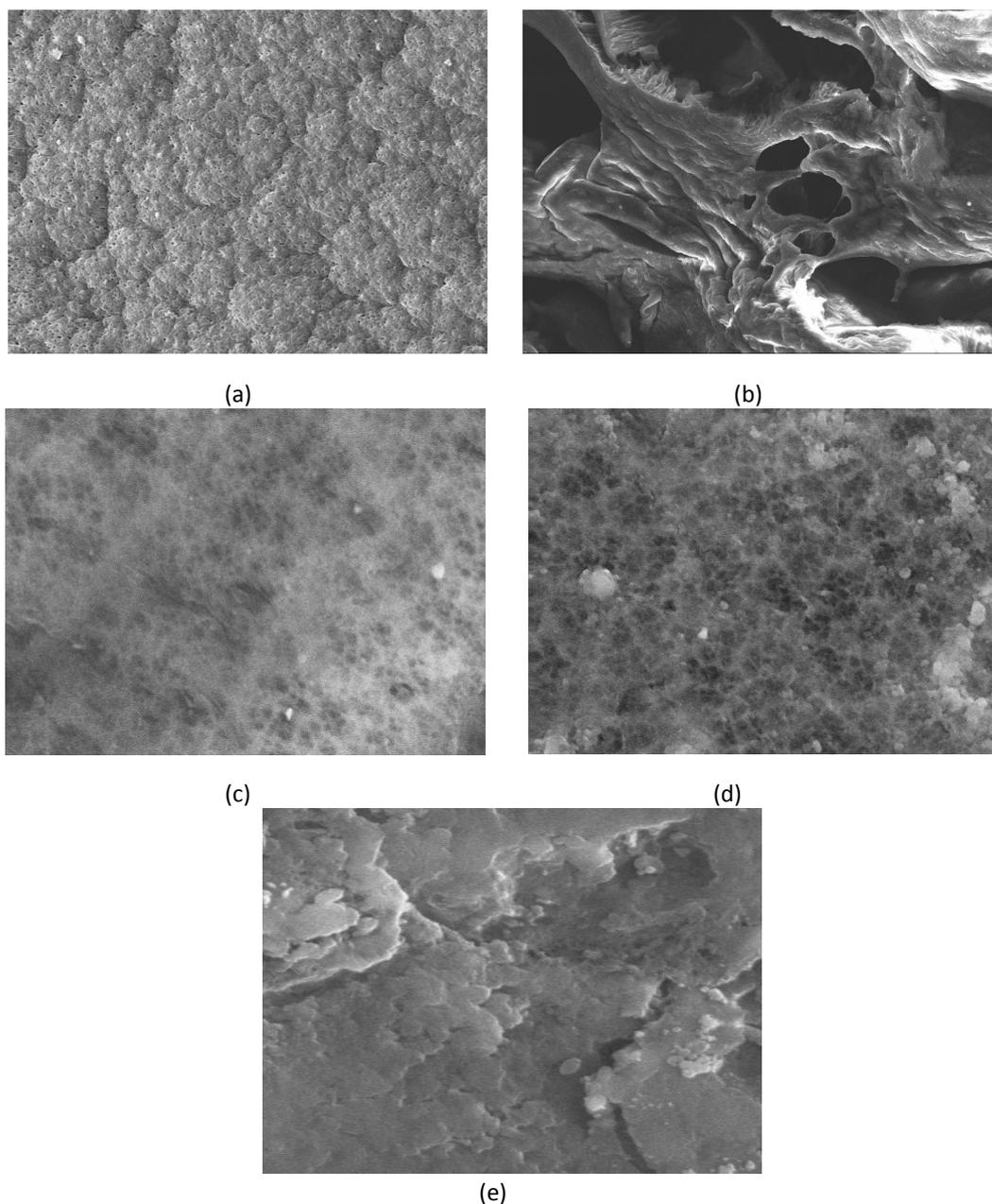


Figure 3: The surface morphology of membrane : (a) PVDF; (b) PSf; dan (c,d,e) PVDF-PSf

To know the effects by an increase in the polysulfone concentration in casting solution and stirring temperature on the morphology structure of PVDF-PSf composite membrane, in the stage of research was conducted analysis of the surface morphology and cross-section of the PVDF-PSf composite membrane using SEM. in figure 3 appear surface morphology analysis results using SEM. Based on the comparison of surface morphology between the PVDF membrane, PSf membrane, and PVDF-PSf membrane composites, it appears that among the trio, the polisulfon membrane have the largest pore size. In addition, based on the figure of 3 (c) and figure 3(e) it appears that the increased levels of PVDF on the casting solution composite membrane structures has resulted, in which up to 5,000 x magnification, the pore on the surface morphology of PVDF-PSf composite membrane is still not appear.

In addition to casting solution composition, based on the results of the SEM analysis that has been done, it appears that temperature stirring also play an important role in determining the surface morphology of the PVDF-PSf composite membrane. This condition appears in figure 3 (b) and 3 (c), where the PVDF-PSf

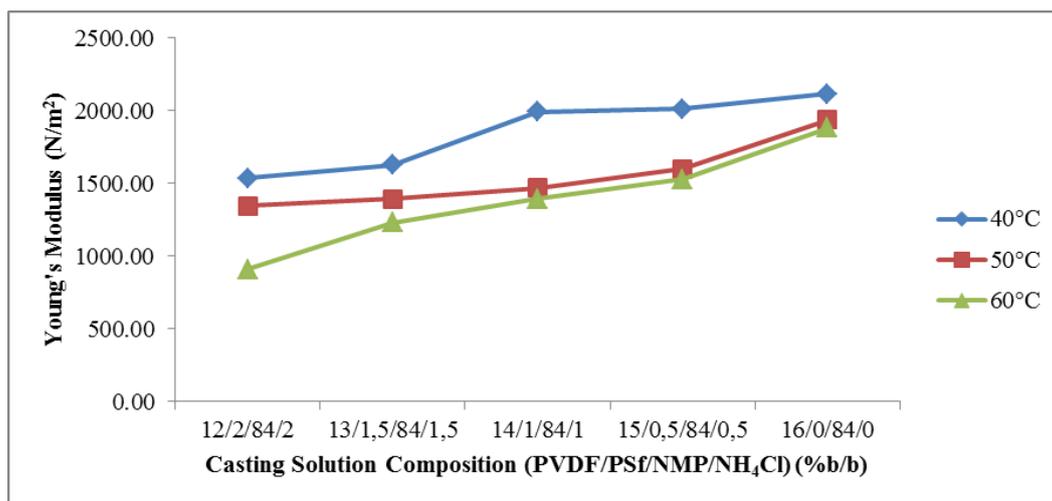
composite membrane resulting from the preparation process by using a higher temperature stirring, will produce a more porous pore structure and observed at 5,000 x magnification.

**The mechanical strength test of PVDF-PSf composite membrane using autograph**

Results of the autographs measurement give the magnitude of stress and strain which experienced by PVDF-PSf composite membrane over the force of 30 kN. The magnitude of mechanical strength that belongs to the PVDF-PSf composite membrane measured by the young's modulus which is the ratio of the stress to the strain. Then, the Young's modulus is the parameter concerned of the membrane's ability in maintaining the pore size during the driving force applied.

The mechanical strength measuring results of the PVDF-PSf composite membrane using autograf looks at table 1. Based on that data, it appears that PSf blending in materials PVDF have lowered the PVDF membrane mechanical strength, which had acquired the mechanical strength of an average of 1.858, 37 N/m<sup>2</sup>; 1,550 13 N/m<sup>2</sup>; and 1,389, 26 N/m<sup>2</sup>, respectively for the PVDF-PSf composite membrane produced from membrane preparations by using stirring temperature 40 °C; 50 °C; and 60 °C. The each results are 12,26%; 20.08%; and 26,16% lower than mechanical strength of PVDF membrane which unmodified using polysulfone on the same stirring temperature.

In addition, based on such data may also be aware that with the ever increasing levels of PSf blended on the PVDF material, the lower mechanical strength of the PVDF-PSf composite membrane measured from young modulus value obtained. Like for example the PVDF-PSf membrane as the results of stirring using a temperature of 40 °C retrieved the value of young's modulus 2,118.09; 2,012.62; 1,992.60; 1,630.11 and 1,538.43 N/m<sup>2</sup>, respectively for the casting solution composition (PVDF/PSf/NMP/NH<sub>4</sub>Cl)%(b/b) 16/0/0/84; 15/5/84/0.5; 14/1/84/1; 13/1.5/84/1.5; and 12/2/84/2. In the meantime, for the PVDF-PSf membrane resulting from the membrane preparation process with stirring temperature 50 °C, the young's modulus values obtained were lower, i.e. of 1,939.72; 1,599.21; 1,469.44; 1,395.29 and 1,346.99 N/m<sup>2</sup>, respectively for the casting solution composition (PVDF/PSf/NMP/NH<sub>4</sub>Cl)%(b/b) 16/0/0/84; 15/5/84/0.5; 14/1/84/1; 13/1.5/84/1.5; and 12/2/84/2. The higher composition of the PSf as a blending agent of PVDF, the more PSf decomposed between the PVDF polymer particles. The interaction between PSf and PVDF formed through interactions between hydrogen atoms of the methyl group on PSf with fluorine atoms on the PVDF molecule or between the oxygen atoms in the PSf with a hydrogen atom in the PVDF molecule have contributed to the decline in bond energy possessed by a PVDF material, which will have an impact on decreasing the ability of membrane in maintaining the pore size (young's modulus), when applied a particular magnitudes driving force.



**Figure 4: The graph of PVDF-PSf composite membrane mechanical strength data**

Not only the composition of the blending agent (PSf), the stirring temperature is also contributing to determine the mechanical strength of the PVDF-PSf composite membrane. Data on figure 4 shows that the blending process is done with the stirring temperature 60 °C produces PVDF-PSf membrane with a lowest

mechanical strength compared with 2 (two) other methods. This is because the higher temperatures used in the stirring process of the casting solution, will be increasingly heat flow that goes into non solvent, as a result of the the pause time absence between the stirring process with immersion-precipitation that is applied. High temperatures on non solvent, will help to determine the interaction between the PVDF and PSf particles with solvent NMP and porogen NH<sub>4</sub>Cl. The condition is evident from membrane mechanical strength data of 1,538.43; 1,346.99; and 908.49 N/m<sup>2</sup>, respectively for the PVDF-PSf composite membrane with a casting solution composition (PVDF/PSf/NMP/NH<sub>4</sub>Cl) (%w/w) 12/2/84/2 which each produced from the stirring temperature 40 °C, 50 °C and 60 °C.

**Permeability of PVDF-PSf composite membrane**

The PSf levels in the casting solution greatly affects the membrane flux. In this study, the composition of the PVDF-PSf composite membrane casting solution is made on a composition range (PVDF/PSf/NMP/NH<sub>4</sub>Cl) (% w/w) 12/2/84/2 – 16/0/0/84: The relationship of the PSf levels of in the casting solution against the flux values magnitude can be seen in figure 5. On figure 5, it appears that the increased levels of PSf in casting solution, which followed by increased levels of porogen NH<sub>4</sub>Cl, will lead to increased flux of waste. This is because increased levels of PSf and NH<sub>4</sub>Cl in a casting solution will result in forming of polymer configurations distributed in more tenuous compared with PVDF particles configuration in a PVDF membranes casting solution without the addition of PSf. The lower density of the polymer particle configuration, has caused the driving force which needed to be able to skip certain particles penetrate the membrane becomes smaller and the speed of the feed solution across the membrane higher.

In line with this, the deployment of the higher stirring temperature also has led to the formation of polymer particle configurations are more tenuous on the solid PVDF-PSf composite membrane. An increase of the casting solution temperature, has led to a greater heat flow into a non solvent H<sub>2</sub>O in coagulation bath. The existence of the heat flow has led to a rapid non solvent mutual difusity rate on the casting solution, which automatically will also accelerate the precipitation rate of the PVDF-PSf composite membrane casting solution prepared by phase inversion technique with immersion-precipitation. Acceleration of the mutual diffusity rate between non solvent and solvent in a casting solution can be the onset because it has a greater energy availability for termination of the interaction that occurs between PVDF with PVDF, or between PVDF with NMP and NH<sub>4</sub>Cl. Membranes are produced from a relatively fast precipitation events, generally have a morphology structure is a mix between a finger pore and a sponge pore type. While the membrane resulting from a relatively slow precipitation event generally have morphology structure with a uniform sponge pore structure of on his cross section.

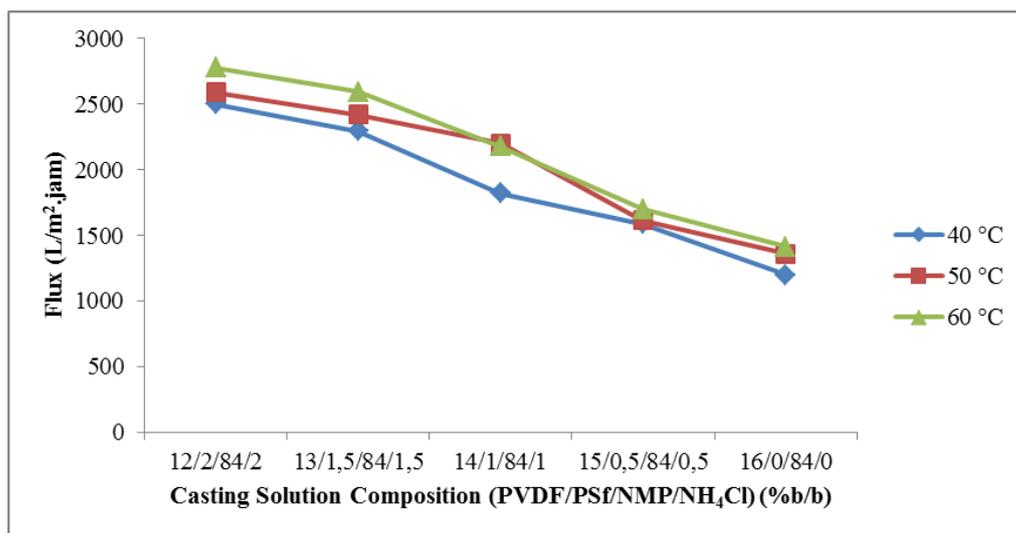


Figure 5: The graph of PVDF-PSf composite membrane permeability data

In figure 5, it appears the data flux of waste from each of the PVDF-PSf composite membrane with a variety of casting solution composition and stirring temperature, resulting from the precipitation process by

using 100% H<sub>2</sub>O. On the casting solution composition (PVDF/PSf/NMP/NH<sub>4</sub>Cl) (%w/w) 16/0/84/0 resulting from the stirring process using a temperature of 40 °C, obtained flux 1,200.38 L/m<sup>2</sup>.h. The waste flux values begin to rise when starting PSf added into the membrane casting solution, where on the PSf addition of 0.5% (w/w) into the casting solution, has obtained the flux membrane of 1,586.29 L/m<sup>2</sup>.h. Further increases in flux occurs when the PSf levels in casting solution enhanced reaches 1% (w/w)-2% (w/w), each of which produces a flux value 1,819.40 L/m<sup>2</sup>.h and 2,295.33 L/m<sup>2</sup> h. In line with that condition, the highest flux, i.e. of 2,499.21 L/m<sup>2</sup>.h obtained when the PSf amount was increased to reach the 2% (w/w).

The higher value on membrane flux are obtained when the PVDF-PSf composite membrane which used in separation, homogenized through stirring at the temperature of 50 °C. An increase in the flux compared to the flux produced by PVDF-PSf composite membrane produced from the casting solution stirring process at a temperature of 40 °C is due to the deployment of higher stirring temperature on the casting solution has led to larger impairment of thermodynamic equilibrium reaction. These disorders are caused due to the availability of thermal energy in the non solvent, which can simplify the termination of the interaction that occurs between the PVDF with NMP and NH<sub>4</sub>Cl. Based on the data in figure 5, the flux values obtained for the PVDF-PSf composite membrane produced from the stirring process using a temperature of 50 °C, each of which is 1,359.07 L/m<sup>2</sup>.h; 1,613.19 L/m<sup>2</sup>.h; 2,200.56 L/m<sup>2</sup>.h; 2,422.12 L/m<sup>2</sup>.h and 2,589.54 L/m<sup>2</sup>.h to casting solution composition (PVDF/PSf/NMP/NH<sub>4</sub>Cl) (%w/w) 16/0/0/84; 15/5/84/0.5; 14/1/84/1; 13/1.5/84/1.5; and 12/2/84/2. In line with this, the highest membrane flux value is obtained from the casting solution which using a stirring temperature of 60 °C.

**Selectivity of PVDF-PSf composite membrane**

Selectivity is one of the membrane ability parameters on hold certain species and skip the other species, which are strongly affected by interaction between speci with membrane interface, size of the species and membrane pore size. The parameters used to determine the selectivity of a membrane is called a rejection coefficient, which is a concentration of dissolved substances which does not penetrate the membrane. On the rejection coefficient measurement of the PVDF-PSf composite membrane, which measured is COD levels of batik waste before and after passed the membrane.

In figure 6, it appears that in the outline of the PVDF-PSf composite membrane selectivity test result against aqueous wastes in contrast with the results of the permeability test. In figure 6, it appears that the PVDF-PSf composite membrane resulting from the phase inversion process with a casting solution composition of PVDF/PSf/NMP/NH<sub>4</sub>Cl (% b/b) 16/0/0/84 and stirring temperature 40 °C, obtained the highest level of membranes rejection. On the phase inversion process of the PVDF-PSf composite membrane using casting solution composition and stirring temperature above, obtained rejection coefficients of 92,78%. The rejection coefficient gradually began to decline when the composition of the PVDF in casting solution is reduced. At present levels of PVDF in the casting solution was reduced to 15% (b/b), the rejection coefficient obtained 92,02%. Further decline occurs when the levels of PVDF on casting solution 14 – 13% (b/b), each of which yielded rejection coefficients of 91,65%; and 91,21%. In line with that condition, the lowest rejection coefficient, i.e. 90,44%, obtained when the levels of PVDF on casting solution only 12%.

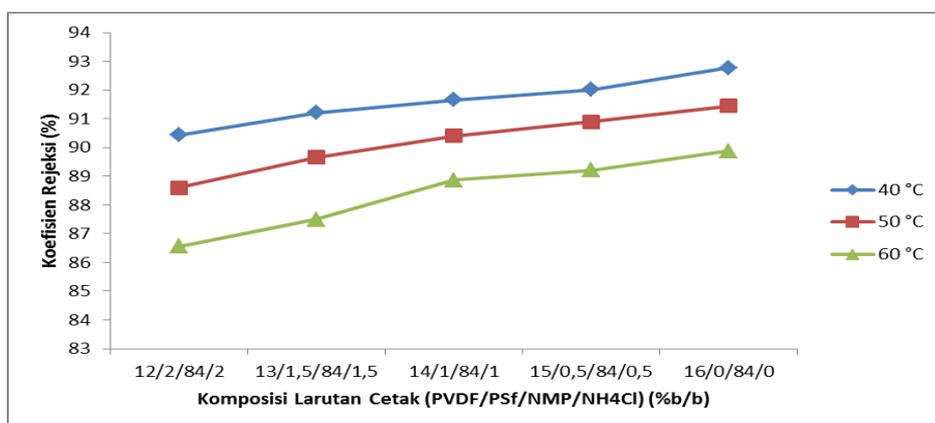


Figure 6: Selectivity (rejection coefficient) data of the PVDF-PSf composite membrane against wastewater

The decrease of PVDF levels in casting solution, will result decrease in the rejection coefficient towards a wastewater. This is because the decrease of PVDF levels in the casting solution, which is accompanied by increasing levels of PSf and porogen on the preparation process of the PVDF membrane, will lead to the formation of the PVDF-PSf polymer configuration which distributed more tenuous, with higher levels of porosity. The low density level of PVDF-PSf polymer particle configurations, has led to the detention of the PVDF-PSf composite membrane against particles in batik wastewater becomes low, when it penetrates the membrane. The decreased of membrane resistance against wastewater causing no longer need large forces to be able to skip the waste particles penetrate the PVDF-PSf composites membrane. Learn more about selectivity data of the PVDF-PSf composite membrane against batik wastewater looks in figure 6.

In line with this, the lower temperature stirring, has also led to the formation of polymer particle configurations more meetings on the solid PVDF-PSf composite membrane. And as a result is needed greater driving force to be able to skip the wastewater particles on porous PVDF-PSf composite membrane. It corresponds to the PVDF-PSf composite membrane selectivity measurement results composite against batik wastewater solution, which when used stirring temperature 60 °C, the rejection coefficient was low, that is obtained by 86,56%. These values gradually increased, along with declining temperature stirring are used, as it appears on the use of stirring temperature 50 °C, obtained rejection coefficients 88,60% for casting solution composition of PVDF/NMP/NH<sub>4</sub>Cl (% b/b) 12/2/84/2. While in use of stirring temperature 40 °C, obtained rejection coefficients of 90,44%.

### CONCLUSION

Based on the results obtained, it can be drawn the conclusion, namely: (1) results of the morphology analysis of the PVDF-PSf composite membrane surface using SEM showed that the casting solution affect the morphological structure of the membrane; (b) results of the surface morphology analysis of the PVDF-PSf composite membrane using SEM showed that the stirring temperature effect on morphological structure of the membrane; (c) casting solution composition and stirring temperature effect on mechanical strength of the PVDF- PSf composite membrane. The mechanical strength of the PVDF-PSf composite membrane is in the range of 908.49 N/m<sup>2</sup> – 2,118.09 N/m<sup>2</sup>; (d) casting solution composition and stirring temperature effect on permeability of the membrane PVDF-PSf composite membrane. The permeability of the PVDF-PSf composite membrane in the processing of wastewater samples are at range 1,200.38 L/ m<sup>2</sup>.h – 2,779.45 L/m<sup>2</sup>.h; (e) casting solution composition and stirring temperature effect on the PVDF-PSf composite membrane selectivity. The rejection coefficients of the PVDF-PSf composite membrane is in the range of 89.88%-92.78%.

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### REFERENCES

- [1] Ki KS, Lee KH, Cho K, Park CE. J Membr Sci 2002;199: 135-145.
- [2] Nunnes SP, Peinemann KV. Membrane Technology in The Chemical Industry. WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim, Germany, 2006.
- [3] Voicu SI, Aldea F, Radut M, Nechifor G. UPB Sci Bull Series B,2008;70:3.
- [4] Elsevier. Comprehensive Membrane Science and Engineering. Elsevier. USA. 2010.
- [5] Grasselli M, Betz N. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms 2005;236 (1-4): 501-507.
- [6] Hashim NA, Liu F, Li K. J Membr Sci 2009;345 (1-2): 134-141.
- [7] Mulder M. The Use of Membrane Process in Water Purification. Proceeding. Bandung, 1996.
- [8] Liu F, Hashim NA, Liu Y, Abed MRM, Li K. J Membr Sci 2011;375: 1-27.